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CEMENT MANUFACTURE

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Long-period Cement Tests.

THE results to date of cement tests commenced in 1916 by the American Portland Cement Association are given in Figs. 1 and 2, which show how the strength characteristics of cement have changed since the year 1916.

Fig. 1 gives the essential data on the progressive increase of strength in cement made during the sixteen-year period. The left-hand diagram in Fig. 1, which is plotted to arithmetical scale, shows the advance in the strength of cements made during the past decade compared with those made prior to 1920. Further detail

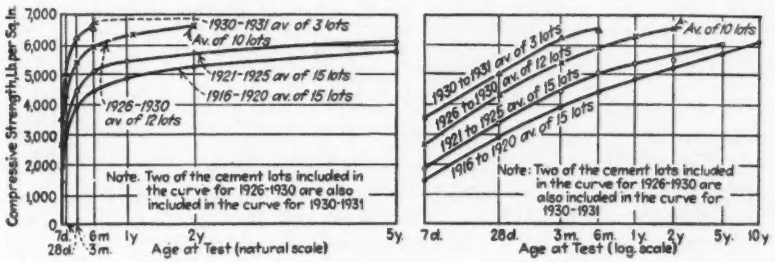
Compressive Strength, Lb. per Sq. In.
7,000
6,000
5,000
4,000
3,000
2,000
1,000
0
1930-1931 av. of 3 lots
Av. of 10 lots
1921-1925 av. of 15 lots
1916-1920 av. of 15 lots
1926-1930 av. of 12 lots
Note. Two of the cement lots included in the curve for 1926-1930 are also included in the curve for 1930-1931
7d. 28d. 6m 3m. 1y 2y 5y.
Age at Test (natural scale)
1930 to 1931 av. of 3 lots
1926 to 1930 av. of 12 lots
1921 to 1925 av. of 15 lots
1916 to 1920 av. of 15 lots
Av. of 10 lots
Note: Two of the cement lots included in the curve for 1926-1930 are also included in the curve for 1930-1931
7d. 28d. 3m. 6m. 1y. 2y 5y. 10y
Age at Test (log. scale)

Fig. 1.

is shown in the right-hand diagram, in which time is plotted to logarithmic scale. It will be seen that the rate of gain in strength is about the same for all the groups but the 1916-1920 and 1921-1925 cements show a continuous increase for periods of as much as ten years, while the two later cement groups give evidence of tending toward a maximum in strength at earlier periods.

It is worthy of special note that the upper curve, for cements made in 1930 and 1931, represents the influence of the strength increase provided by the standard cement specification adopted by the American Society for Testing Materials in

1930. Chemical analyses and fineness measurements indicate that the high early strength of the later cements is due in part to changes in chemical composition and in part to greater fineness, though the two separate effects could not be isolated. That these characteristics of concrete strength differ greatly from the strength relations of mortar specimens is shown by Fig. 2, representing concrete cylinder tests compared with mortar cylinder (compressive) and briquette (tensile) tests on cements of the 1916 to 1920 group, tested at various times up to ten years' average age. The mortar specimens in both tension and compression tests reached their maximum after three to six months, and then slightly declined or remained at a constant value, while the concrete specimens gained in strength almost uniformly throughout the ten-year period. In all cases the cements included in the tests were mixtures of four or five commercial brands bought from local dealers. The tests were begun in 1916 at Lewis Institute, Chicago, and have been continued ever since. Corresponding tests of specimens stored dry have apparently not been made.

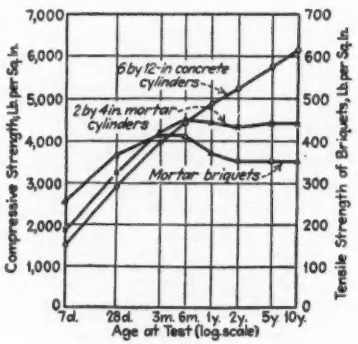

Fig. 2.

In Fig. 1 the dates noted on the curve represent the period during which the cement was bought. All tests were made on 6-in. by 12-in. cylinders using a 1 : 5 mix, with aggregates up to 1½ in. in size. Specimens were cured in moist air until the time of test. There is a progressive increase of strength between successive five-year periods and also a long-continued rise in strength, the earlier cements showing a steady gain up to the ten-year period represented. The results are shown on both natural and logarithmic abscissa scales to emphasise the strength-time relation. The logarithmic plot reveals practical uniformity in the rate of increase of strength.

The curves in Fig. 2 show that concrete gains in strength while mortar specimens in compression and tension reach a maximum at three months. These tests, showing the lack of correlation between cement strength and concrete strength, were made on cement purchased during the five years 1916 to 1920. The mortar specimens, of 1 : 3 mix, were cured in water until test, and were tested damp; the data for the concrete specimens are as given in Fig. 1.

Heat Transmission in Rotary Kilns.—III.

By Wm. GILBERT, Wh.Sc., M.Inst.C.E.

(44) The total quantity of heat which can be transmitted to the material in a rotary kiln is obtained by calculating the rate of heat transmission in a number of typical rings each of which is formed by two cross sections of the kiln shell taken 1 ft. apart.

The method can be fully explained by reference to two rings only, one in the zone of flame radiation and one in the zone of gas radiation and convection. It is therefore proposed to work out in detail the rate of heat transfer in two such rings before applying the method generally to kilns of various diameters and lengths.

The Cosine Law.

(45) In the kiln a large part of the heat is first transmitted to the firebrick lining and then radiated from it to the material, hence reference is made to the laws which govern radiation under such conditions.

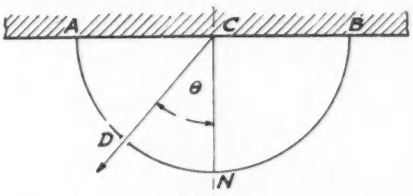

Fig. 6.

Consider a plane surface of hot firebrick such as that shown at *ACB* in Fig. 6. A hemisphere is described about the centre *C*, and *CN* is the normal at *C*. Heat radiated from a small portion of firebrick at *C* will pass through the surface of the hemisphere at all points, and the quantity which emerges through any small area at *D* will be proportional to that area multiplied by the cosine of the angle *DCN*. The total heat radiated can be summed up over the surface of the hemisphere, and scale is obtained by remembering that the same total will be given by Table I. Fig. 6 is introduced to show how the relative intensity of the radiation in any direction can be estimated.

(46) Next let Fig. 7 represent part of a longitudinal section of a rotary kiln, the material layer being at *M*. The tube is shown divided into annular rings, numbered 1, 2, 3, 4, etc., by cross sections spaced 1 ft. apart. Another view of ring No. 1 is shown in Fig. 8. It is assumed that the inside surface temperature of the firebrick lining varies uniformly and not too rapidly from ring to ring in Fig. 7. Attention is concentrated on 1 square foot of the lining in the roof of each ring, as shown for instance at *ab* in Figs. 7 and 8. The radiation from *ab* in ring No. 1 is sent out in all directions up and down inside the kiln in accordance with the cosine law, and it is apparent that the portion of it which falls on ring No. 2,

for instance, will be balanced by the heat radiated into ring No. 1 from 1 square foot of the lining at *da* in the roof of ring No. 3. Similarly, the radiation from *ab* which falls in ring No. 4 will be replaced in ring No. 1 by the radiation from *fd* in ring No. 5.

For calculation purposes we may therefore treat the whole of the radiation which leaves any small area of the firebrick surface in ring No. 1 as if it remained entirely in that ring, or, in other words, as if all the radiation took place in the plane of the paper in Fig. 8. It can also be proved mathematically that the cosine law will apply.

(47) We may next consider the manner in which heat is radiated from the lining to the material chord in ring No. 1 as shown by Fig. 8. The lining surface temperature is assumed to be uniform throughout the arc *AEFB*. Take a small

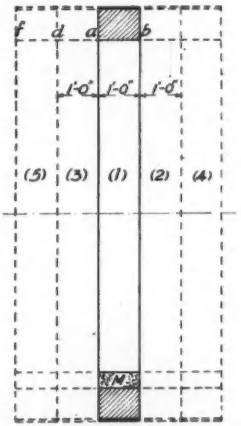

Fig. 7.

area at *E* on the surface of the lining and draw lines *EA* and *EB* to each end of the material chord. *EF* is a diameter of the circle. The intensity of the radiation in the direction *EB* is proportional to cos θ, and since all the radiation is in the plane of the paper it may easily be proved that the total radiation sent out between the normal *EF* and the line *EB* is proportional to sin θ. To the same scale the total radiation leaving the surface at *E* which takes place over one right angle on each side of the normal will be 2 sin 90 deg. = 2·0. Hence the fraction of the total radiation from the small area at *E* which reaches the material chord is

$$\frac{\sin(\phi + \theta) . - \sin\theta}{2 .}$$

In the position shown in Fig. 8 $\phi = 45$ deg., and $\theta = 7$ deg., so that the fraction becomes

$$\frac{\sin 52 \text{ deg.} - \sin 7 \text{ deg.}}{2} = 0{\cdot}333$$

If the calculation is made at a number of points equally spaced around the arc *AEFB* for a charge of 9 per cent., it will be found that on the average 0·298 of the total radiation which leaves the firebrick lining will fall on to the chord *AB*. But the ratio of the length of the chord *AB* to the arc *AEFB* is also found to be 0·298. From a diagram drawn to scale the radiation received by the chord is also found to be spread uniformly over the surface, hence, for the purpose of calculating the heat exchange by radiation, the lining surface and the chord surface may be treated as parallel and adjacent plates.

Ring No. 1 in Flame Radiation Zone.

(48) The ring selected for illustration is taken in the enlarged zone of the kiln described in the December, 1932, number of this journal, and at a point where three-quarters of the $CaCO_3$ has been decomposed, that is at 83·5 per cent. of the length on Fig. 2. From a model, a charge of 6 per cent. in the drying

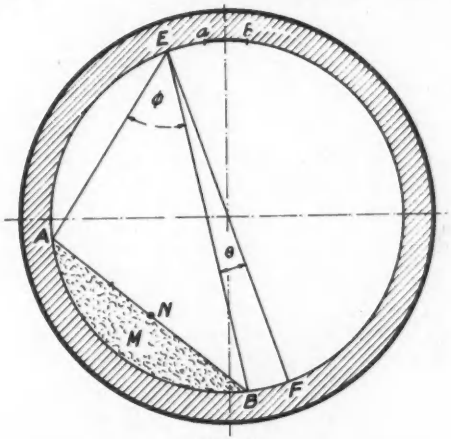

Fig. 8.

portion of the kiln was found to correspond to a charge of 9 per cent. in the enlarged zone, the outlet from the latter being contracted.

The following particulars are required for calculation purposes:

(*a*) Flame temperature .. .. 2,450 deg. F.
(*b*) Material temperature .. .. 1,600 deg. F.
(*c*) Charge per cent. .. .. 9·0
(*d*) Kiln diameter inside lining .. .. .. 8·5 ft.
(*e*) Length of upper arc *AEFB* in Fig. 8 .. .. 20·1 ft.
(*f*) Length of material arc *ACB* .. .. .. 6·6 ft.
(*g*) Length of material chord *AB* .. .. .. 6·0 ft.
(*h*) A point on the kiln lining traverses the upper arc in 0·793 minutes, and the lower arc in 0·26 minutes, total 1·053 minutes.
(*i*) The emission or absorption factors used for the various surfaces are: coke particles 0·90, kiln lining 0·90, material chord 0·85, and material arc 0·75.
(*j*) The density factor *F* for the coke particles is 0·90.

(49) Further to explain the value given for F in line (*j*) reference may be made to Fig. 9 which shows a small tube built into the combustion zone along a diameter. The size and pitch of the coke particles are similar inside and outside the tube.

If a parallel ray of heat or light is supposed to pass through the tube, in this case the probability is that 90 per cent. of it would be intercepted and 10 per cent. would pass through. It is seen, therefore, that the value of F in formula (2) (which may be called the density factor) depends on the size and pitch of the coke particles and not on the radiating power of their surfaces.

When the volatile matter is driven off and coke particles are formed it is generally assumed that some shrinkage occurs. For this reason values calculated for F by formula (2) may be somewhat too large, and an allowance is made by taking the rather low value of 0·9 for the emission factor of incandescent carbon.

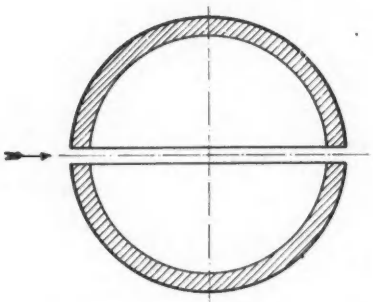

Fig. 9.

(50) Returning to ring No. (1) as shown in Figs. 7 and 8, it should be noted that under normal working conditions the material passing through the ring will always have the same average temperature, and the problem is to find how much heat can be transmitted to it per minute, either directly by radiation from the flame or indirectly by radiation from the kiln lining. At the ring considered the $CaCO_3$ is being decomposed, and the material temperature would rise about 16 deg. F. during its passage through the ring.

On commencing the calculation the temperature of the inside surface of the firebrick lining has to be assumed. It is taken at 2,285 deg. F. The values of the black body radiation in B.T.U.'s per square foot per minute for the three temperatures used are, from Table I:

	Temperature. Deg. F.	Black body radiation.
Flame	2,450	2,070
Lining	2,285	1,640
Material	1,600	520

(51) Details of the heat transmission in the ring, which is 1 ft. wide, are as follows :

	B.T.U.'s per minute.
(*a*) Radiation from the flame to the firebrick lining in the upper arc, *AEFB*, in Fig. 8, $(2{,}070 - 1{,}640) \times 0{\cdot}9 \times 0{\cdot}9 \times 0{\cdot}9 \times 20{\cdot}1$ ..	6,299
(*b*) Radiation from the flame to the material chord *AB*, $(2{,}070 - 520) \times 0{\cdot}9 \times 0{\cdot}9 \times 0{\cdot}85 \times 6{\cdot}0$ ..	6,401
(*c*) Radiation from the lining of the upper arc to the material chord *AB*, $(1{,}640 - 520) \times 0{\cdot}10 \times 0{\cdot}9 \times 0{\cdot}85 \times 6{\cdot}0$ .. (The factor 0·10 is explained later.)	514
(*d*) Radiation from the firebrick lining in the material arc *ACB* to the material, $(1{,}640 - 520) \times 0{\cdot}9 \times 0{\cdot}75 \times 6{\cdot}6$	4,989

(52) To obtain the value of $F = 0{\cdot}9$ as used in lines (*a*) and (*b*) the value of *NaD* was calculated by formula (3), the details being :

$$NaD = \frac{2{,}219 \times 7{\cdot}6}{7 \times 867}\left(\frac{75{\cdot}8}{100}\right)^{\frac{2}{3}} = 2{\cdot}32$$

Hence *F*, from Table V, = 0·90. It should be noted that owing to the 9 per cent. charge the mean value of *D* the kiln diameter was taken at 7·6 instead of 8·5 ft.

In line (*c*) the radiation from the lining to the chord has to pass through the coke particles which intercept 0·9 of it, hence the fraction reaching the chord is 1·0 — 0·9, or 0·10.

In line (*d*) the material will mainly rest on the lining in the form of small lumps and, the area of contact being small, heat is assumed to be transmitted by radiation. The surface temperature of the lumps may rise more quickly than their average temperature if they are stationary on the lining, and the rate of heat transfer is then reduced. Hence the rather low absorption factor of 0·75 is used.

It will be realised that the various factors listed in para. (48) *i* have been selected after many trials because they best fit the results which have been obtained from tests on various kilns in practice.

(53) Returning to the calculations in para. (51), the heat transmitted to the material is found by adding the quantities in lines (*b*), (*c*) and (*d*), the result being 11,904. It remains to take account of the loss of heat by radiation from the kiln shell. It is found by an examination of several kilns that the shell radiation loss from any ring 1 ft. wide is a reasonably constant fraction of the heat transmitted to the material in the ring. In the kiln under consideration the ratio was 0·068. Hence the shell radiation loss in ring No. 1 is $0{\cdot}068 \times 11{,}904 = 809$ B.T.U.'s. This quantity of heat is included in line (*a*), and the remainder must balance the heat taken from the lining in lines (*c*) and (*d*), or

6,299 — 809 must equal 514 + 4,989.

The agreement in this case is satisfactory, and it shows that the surface temperature of the firebrick lining was correctly assumed at 2,285 deg. F.

(54) The heat quantity in para. (51) line (*c*) is radiated from the lining to the material chord as it is received from the flame, but the heat quantity of 4,989 B.T.U.'s in line (*d*) is stored and then withdrawn from the lining as it passes under the material. It is necessary to ascertain if this can be done.

The problem is best considered in relation to a single lining block the inner face of which is 1 ft. square. From para. (48) *h* such a block would receive heat for 0·793 minute and reject heat for 0·26 minute during each revolution or cycle, and since the lining speed is 25·34 ft. per minute the heat stored and given up will be $\frac{4,989}{25·34}$ or 197 B.T.U.'s.

Referring to Figs. 10 and 11 which represent the conditions, the face *EF* of the block is assumed to receive heat from a screen *AB* at a temperature of 2,450 deg. F. during the first period, and to reject heat to the screen *CD*, which is at a temperature of 1,600 deg. F. during the second period.

(55) The surface temperature of the block will not remain constant, since it will rise when heat is being taken in and fall when heat is being rejected, and it is necessary to assume for calculation purposes that the rate of heat supply to and from the block is proportional to the temperature difference available.

Let *R* be the rate of heat supply from the block to the screen *CD* expressed in B.T.U.'s per degree F. temperature difference per minute. Since the average temperature difference is 685 deg. F. we have

$$R \times 0·26 \times 685 = 194. \quad \text{Hence } R = 1·106.$$

The same quantity of heat is supplied from the screen *AB* to the face of the block during each cycle, but the time is greater and the temperature difference is less.

Putting *Q* for the rate of heat supply to the block per degree F. temperature difference per minute, we have

$$Q \times 0·793 \times 165 = 197. \quad \text{Hence } Q = 1·505.$$

(56) A Fourier Series is now used to calculate the fluctuation of the surface temperature, and also the temperature distribution inside the block during each cycle, or kiln revolution, and two graphs have been drawn to show the results obtained. In Fig. 12 time is measured horizontally and the surface temperature of the block at corresponding periods is shown vertically. The graph commences at the period when the screen *AB* in Fig. 10 begins to radiate heat to the face of the block, and the surface temperature is seen to rise from 1,970 deg. to 2,388 deg. F. during the period of 0·793 minute. The block then begins to reject heat to the screen *CD* as shown in Fig. 11 and the surface temperature falls to 1,970 deg. F. at the end of the cycle.

The original assumption that the surface temperature of the kiln lining remained constant during a revolution, although necessary, was not correct. From the graph the average surface temperature of the block for the complete cycle is seen to be 2,267 deg. F., but during the period of receiving heat the average

surface temperature was 2,336 deg. F. and during the period of rejecting heat the average surface temperature was 2,072 deg. F.

(57) Taking into account the reduced temperature difference available we now have

(*a*) Heat entering the block per cycle
$$= 1{\cdot}505 \times (2{,}450 - 2{,}336) \times 0{\cdot}793 = 136 \text{ B.T.U.'s.}$$
(*b*) Heat leaving the block per cycle
$$= 1{\cdot}106 \times (2{,}072 - 1{,}600) \times 0{\cdot}26 = 136 \text{ B.T.U.'s.}$$

The reduced quantity is $\frac{136}{197} \times 100 = 69$ per cent. of the original estimate, see para. (54).

(58) Referring again to the conditions shown in Figs. 10 and 11, Fig. 13 shows the temperature distribution at various depths below the surface of the block. The lower curve relates to the instant when the block begins to receive heat from the screen *AB* and the upper curve to the instant when the block commences to reject heat to the screen *CD*. It is seen that at a distance of 0·30 in. below the surface the temperature fluctuation has practically disappeared.

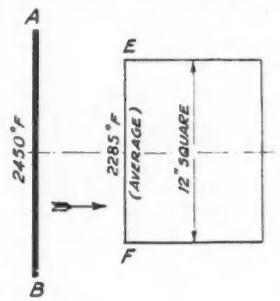

Fig. 10.

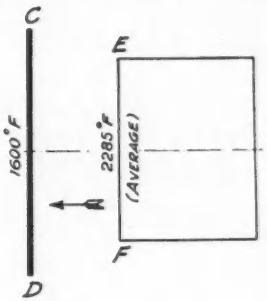

Fig. 11.

Confirmation of the quantity of heat which enters or leaves the block per cycle may be obtained from Fig. 13 as follows. The weight of a slab of firebrick, 1 square foot in area and 0·30 in. thick, at 137 lb. per cubic foot, is
$$\frac{137 \times 0{\cdot}3}{12} = 3{\cdot}43 \text{ lb.}$$

From the two curves on Fig. 13 the average temperature fluctuation over the depth of 0·3 in. is 152 deg. F., and since the specific heat of the firebrick is 0·26 the heat stored per cycle is
$$3{\cdot}43 \times 152 \times 0{\cdot}26 = 136 \text{ B.T.U.'s, as given in para. (57).}$$

The conductivity of the firebrick at the inner surface of the lining at the temperature range considered is 11·15 B.T.U.'s per square foot per hour per deg. F. per inch thickness.

(59) It should be mentioned that the calculations of heat conduction which were made by the use of a Fourier Series, and on which the graphs in Figs. 12 and 13 are based, proved to be exceptionally difficult. It was necessary to assume

that the rate of heat transfer to and from the firebrick block was proportional at any instant to the temperature difference, and therefore not in accordance with the fourth power law, but if the constants Q and R in para. (55) are correctly obtained the error in the final result will be small.

(60) The next step is to revise the calculations given in para. (51). A reducing factor of 0·69 is introduced into lines (*a*) and (*d*) to allow for the reduced temperature difference available, and an allowance is made in line (*c*) where the average temperature difference is somewhat increased. In order to obtain a balance between the heat taken from and given to the upper lining arc per minute, it is necessary

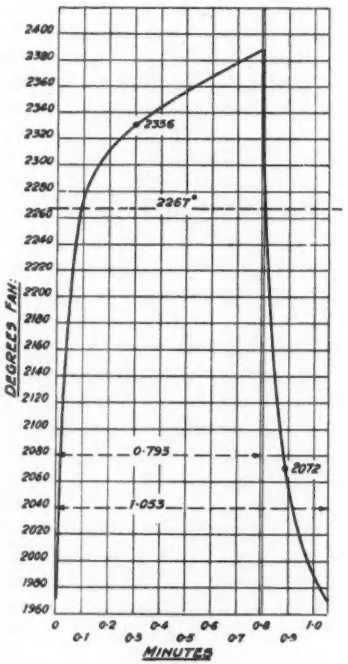

Fig. 12.

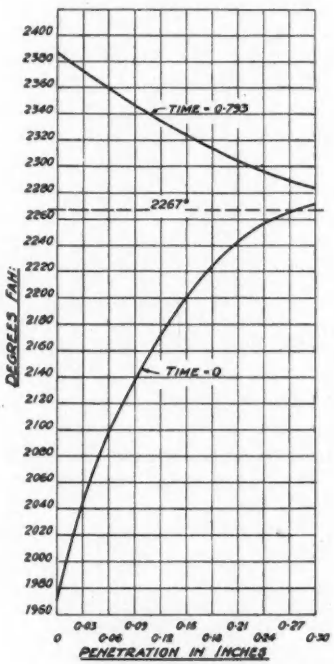

Fig. 13.

slightly to change the average lining temperature throughout the revolution which now becomes 2,274 deg. F. The corresponding black body radiation from *Table I* is 1,614.

The revised calculations for ring No. 1 are now as follows:

	B.T.U.'s per minute.
(*a*) Flame to firebrick lining, $456 \times 0{\cdot}9 \times 0{\cdot}9 \times 0{\cdot}9 \times 0{\cdot}69 \times 20{\cdot}1$	4,609
(*b*) Flame to material chord, $1{,}550 \times 0{\cdot}9 \times 0{\cdot}9 \times 0{\cdot}85 \times 6{\cdot}0$	6,401

(*c*) Firebrick lining to material chord,

$$1{,}094 \times 0{\cdot}10 \times 0{\cdot}9 \times 0{\cdot}85 \times 6 \times \frac{729}{674} \quad .. \quad .. \quad 543$$

(*d*) Firebrick lining to material arc,

$$1{,}094 \times 0{\cdot}9 \times 0{\cdot}75 \times 0{\cdot}69 \times 6{\cdot}6 \quad .. \quad .. \quad .. \quad 3{,}362$$

Adding lines (*b*), (*c*) and (*d*) we have

	B.T.U.'s.
Heat given to material..	10,306
(*e*) Add for shell radiation 10,306 × 0·068	701
Total heat transfer in ring	11,007

The heat taken from the upper lining arc per minute is the sum of the quantities in lines (*c*), (*d*), and (*e*), or 543 + 3,362 + 701 = 4,606, and this corresponds to the heat supplied to the upper lining arc per minute, see line (*a*).

This ring 1 ft. wide is assumed to represent the length of the kiln in which one half of the $CaCO_3$ is decomposed. The heat required for this purpose, including the shell radiation loss, is found from the heat balance to be 132,310 B.T.U.'s at the normal output of 6·97 tons of clinker per hour. Hence the corresponding length in the kiln is

$$\frac{132{,}310}{11{,}007} = 12{\cdot}0 \text{ ft.}$$

(61) The preceding calculation of a typical ring in the combustion zone can be considerably shortened when the details are understood. For instance, the reducing factor used in connection with the heat stored in the firebrick lining has been calculated and tabulated for a sufficient number of rings throughout a kiln, and the figures, with small adjustment, can be used for other kilns. This factor being known in the first instance the calculation of the heat transfer in any ring need only be made once.

If this method of calculating the rate of heat transfer from flame to material in a typical ring is accepted as satisfactory, a quantity of useful information becomes available. For instance the calculations which relate to ring No. 1 can be repeated for a range of diameters and material charges in order to determine the best dimensions for the clinkering zone and for the material charge at any given output. Such information it is hoped to give later.

Ring No. 2.

(62) The second ring on which the typical calculations are made occurs in the drying zone. It is taken at 45 per cent. of the kiln length in Fig. 2, of Part I of this series (December, 1932), and in this position three-eighths of the slurry moisture has been evaporated. In the drying zone, see para. (7), the temperature of the inner surface of the firebrick lining can be approximately calculated from the shell temperature as measured and from the thickness and the conductivity of the firebrick lining. In individual cases the calculation may be in error owing to various causes, and it has therefore been necessary to make the investigation on several kilns. In two cases at least (wet-process kilns from 200 to 250 ft.

long) the conditions appear to have been favourable throughout. The average result is shown approximately by the curve *X W V* on Fig. 2.

(63) Proceeding with ring No. 2, the figures which follow are required for calculation purposes:

(*a*) Gas temperature 1,496 deg. F.; lining surface temperature inside, 660 deg. F.; material temperature, 212 deg. F.

(*b*) Kiln.—Diameter inside lining, 7·65ft.; charge, 6 per cent.; upper arc, 18·85 ft.; chord, 4·79 ft.; lower arc, 5·15 ft.

(*c*) Emission or absorption factors.—Firebrick lining, 0·80; material chord, 0·80. These values are used in the drying zone where the slurry is wet or only partly dry on the surface.

(*d*) Convection constant H_c, in B.T.U.'s per square foot per hour per deg. Fah. Gas to lining, 1·76; gas to material, 1·87.

(*e*) Values of P.D. for gas radiation, refer to this journal for March, 1933 (27), $H_2O = 1{\cdot}90$, $CO_2 = 1{\cdot}20$.

(64) The lining surface temperature fluctuates considerably during a revolution, and in the case of ring No. 2 the average value for the upper arc, which is 725 deg., is taken from a graph to be described later.

The value of the black body radiation and the gas radiation for the three temperatures which occur are tabulated below. The figures are taken from Tables I, II, III, and IV in Part II of this series (March, 1933).

Temperature, deg. Fah.	1,496	725	212
Black body radiation	—	57·0	5·9
Radiation due to H_2O	124·0	20·2	1·9
,, ,, CO_2	42·2	6·2	0·4
Loss due to shielding	10·0	0·4	—
Gas radiation, total	156·2	26·0	2·3

(65) The heat transmitted in the ring can now be set down as follows:

	B.T.U.'s per Minute.
(*a*) Gas radiation to upper arc, $(156{\cdot}2 - 26{\cdot}0) \times 18{\cdot}85 \times 0{\cdot}8$	1,963
(*b*) Gas convection to upper arc, $(1{,}496 \text{ deg.} - 725 \text{ deg.}) \times \frac{1{\cdot}76}{60} \times 18{\cdot}85$	426
(*c*) Gas radiation to material chord, $(156{\cdot}2 - 2{\cdot}3) \times 4{\cdot}79 \times 0{\cdot}80$	590
(*d*) Gas convection to material chord, $(1{,}496 \text{ deg} - 212 \text{ deg.}) \times \frac{1{\cdot}87}{60} \times 4{\cdot}79$	192
Total	3,171

(66) These heat quantities are final, but some further explanation is required. Of the heat transferred to the lining under headings (*a*) and (*b*) it is shown below that 84 B.T.U.'s are radiated from the lining (through the gas) to the material chord and 202 B.T.U.'s are lost in shell radiation. The balance of 2,389 − 286 = 2,103 B.T.U.'s is stored in the firebrick lining and transferred to the underside of the charge. With material damp on the surface the rate of heat transfer is rapid, but it cannot be calculated in this instance and it is not necessary to do so. Since the surface temperature of the lining in the upper arc is known, the amount of heat which can be transferred to it per minute from the gas is also known, and the lining must dispose of the heat which it receives otherwise its average temperatures would rise. The destination of the heat supplied to the lining by the gas is worked out in the following.

(67) HEAT TRANSFER FROM UPPER LINING ARC TO CHORD.—Turning to the Table in para. (64) it is seen that if the upper lining arc at an average temperature of 725 deg. F. is sending out black body radiation, the gas will absorb 26 B.T.U.'s for each square foot of lining surface. If the lining is not sending out black body radiation the gas will absorb proportionately less. The chord will absorb part (in this case 80 per cent.) of the radiation which reaches it, the balance being reflected back and partly absorbed by the gas and partly by the lining.

Approximately the heat radiated from the lining at 725 deg. and retained by the chord is—

	B.T.U.'s.
$(57{\cdot}0 - 26{\cdot}0) \times 0{\cdot}8 \times 0{\cdot}8 \times 4{\cdot}79$ .. .. .. =	95·0
Similarly the radiation from the chord at 212 deg. F. which is ultimately absorbed by the lining is $(5{\cdot}9 - 2{\cdot}3) \times 0{\cdot}8 \times 0{\cdot}8 \times 4{\cdot}79$ .. .. .. =	11·0
Difference	84·0

The quantity in this instance is small, but the method of calculation should be noted.

HEAT LOST BY UPPER ARC IN SHELL RADIATION.—From para. (53) this is seen to be $\frac{0{\cdot}068}{1{\cdot}068} \times 3{,}171 = 202$ B.T.U.'s per minute.

(68) HEAT STORED IN FIREBRICK LINING.—It remains to be seen if the balance of 2,103 B.T.U.'s per minute can be stored in and withdrawn from the firebrick lining. The length of the inside circumference of the lining is $7{\cdot}65\pi$, or 24 ft., and the kiln makes one revolution in 1·053 minutes; hence in one revolution, or in one cycle, each square foot of the lining should transfer to the underside of the material

$$\frac{1{\cdot}053 \times 2{,}103}{24{\cdot}0} = 92{\cdot}3 \text{ B.T.U.'s.}$$

By the aid of a Fourier Series, calculations were made in accordance with the method described in connection with ring No. 1, and from the results obtained the graphs shown in Figs. 14 and 15 have been prepared. It is seen from Fig.

14 that the inside surface temperature of the lining fluctuates during a revolution from 431 deg. to 844 deg. F., the average value for the upper arc being 725 deg. F. as stated in para. 64.

(69) Fig. 15 shows the temperature at various depths below the surface. The lower curve corresponds to time = 0 on Fig. 14 and the upper curve to time = 0·827. Taking a slab of firebrick 1 foot square and 0·24 in. thick, the average temperature fluctuation in the slab, as measured from the graph, is 130 deg. F. Following the method of para. 58 the heat stored per cycle is

$$137 \times \frac{0{\cdot}24}{12} \times 130 \times 0{\cdot}26 = 92{\cdot}6 \text{ B.T.U.'s.}$$

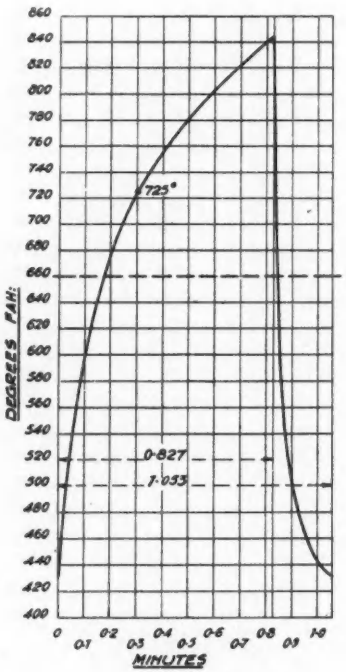

Fig. 14.

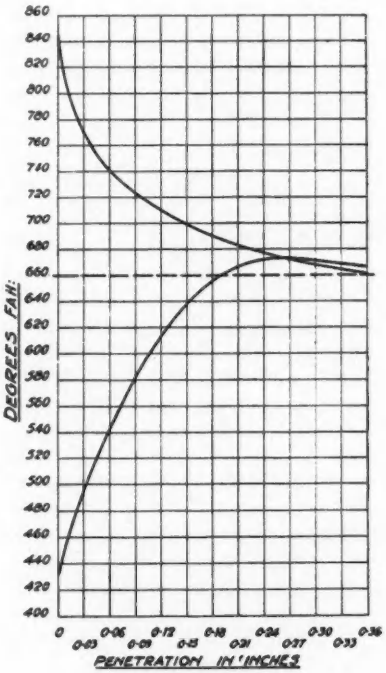

Fig. 15.

This compares with the figure of 92·3 given in para. 68. It is apparent therefore that the lining can store the heat which it receives from the gas and can transfer it to the material arc with a surface temperature fluctuation of about 400 deg. F. The conductivity of the firebrick for the temperature range considered has been taken at 6·60.

(70) At the commencement of the calculations for ring No. 2 the average temperature inside the lining throughout the revolution could be taken from the

curve *XVW* in Fig. 2 as 660 deg. F. The temperature difference between the gas and the lining was then 1,496 — 660 = 836 deg. F. Ultimately the average temperature difference between the gas and the surface of the lining in the upper arc was found to be 1,496 — 725 = 771 deg. F., or a loss of 8 per cent.

This loss is found (by the use of the Fourier Series) to remain approximately constant throughout the drying zone, hence the method adopted when calculating typical rings in this part of the kiln is to take the average surface temperature of the lining from a curve like that shown at *XWV* in Fig. 2 and then to reduce the temperature difference available for the supply of heat from the gas to the lining by 8 per cent.

(71) Ring No. 2 applies to the evaporation of the slurry moisture over the period from a quarter dry to half dry, the heat required for the purpose at an output of 6·97 tons per hour being 81,550 B.T.U.'s per minute. Hence the length of kiln required is

$$\frac{81{,}500}{3{,}171} = 25{\cdot}71 \text{ ft.}$$

This investigation of ring No. 2 applies to a kiln without chains or slurry lifters. It will be seen that the effect of changes in the kiln diameter and in the material charge can be estimated by this method with a reasonable expectation of accuracy. The length of plain tube which would be necessary to reduce the exit gases to any temperature required, and the corresponding output, can also be deduced.

(72) The method of calculation given for ring No. 2 applies throughout the drying zone on the assumption that the firebrick lining is dry on the surface. In the earlier stages, where the surface might remain wet throughout a revolution, the method of calculation is the same but the fluctuation of the surface temperature would be less, thus leading to a somewhat better temperature difference for the supply of heat from the gas to the lining.

(73) The Fourier Series which has been used to work out the temperature fluctuation in the kiln lining, and the quantity of heat which could be stored during each revolution, was devised for the writer by Dr. A. G. Forsdyke, Ph.D., A.R.C.S., D.I.C., after a mathematical investigation which proved both tedious and difficult. The general form of the Series will be given later.

Diesel Excavators.—Messrs. Ruston-Bucyrus, Ltd., of Lincoln, have delivered four of their new type Diesel excavators recently ordered by Stewarts & Lloyds, Ltd., for use in the development of the new steel works which are being erected at Corby. Quick delivery of these machines was essential, and two were sent complete by road. All the excavators are now at work on the site. Two of the machines commenced working on the site approximately three working days after the receipt of the order at Lincoln. Eight Ruston-Bucyrus excavators are employed on this large new enterprise.

The Cement Industry in Russia.

THE progress of the cement industry in Russia as a result of the first Five Years' Plan is reviewed in recent numbers of *Zement*. According to an official publication of the Soviet seven new cement works have been constructed in the five-year period, eighteen works have been renovated and extended, and four works have been renovated. In 1933 a further eight new works should be completed. It is pointed out that in spite of these extensions and renovations the cement output in 1932 was only 3,700,000 tons, as against 6,350,000 tons required by the programme and 5,300,000 tons in 1931. The sum of 222 million roubles was budgeted for the reconstruction of the industry, but 343 million roubles have been expended in the five years.

It was anticipated that one and a half years would suffice to build a cement works, but some works have taken two to three years to build while others are incomplete after four or five years. The supply of constructional materials has been far below requirements. Large quantities of imported plant have not yet been erected, and in many works the most essential repairs have not been possible owing to lack of spare parts. Much of this failure is due to the shortage of workers' dwellings and social organisations and to the low standard of living.

In the official announcements, which it is suggested are greatly exaggerated, an output of 4,700,000 tons is claimed for 1932. It is also stated that in all new and reconstructed works the most modern tube-mills and kilns are installed. The raw slurry is mixed by pneumatic plant and conveyed by eccentric pumps. The cement is conveyed by Fuller-Kinyon pumps. In future all plant will be constructed by the Russian machine industry. There are, however, works using old plant such as Schneider kilns, and the production of these will be increased by 50 to 60 per cent. by using forced draught. Three different cements, in addition to slag and puzzolana cements, are produced. The lowest quality, which frequently scarcely attains a crushing strength of 2,100 lb. per sq. in., comprises 31 per cent. of the output.

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Chemistry of Cement.

By A. C. DAVIS, A.M.I.Mech.E., M.Inst.C.E.I., F.C.S.

WORKS MANAGING DIRECTOR, ASSOCIATED PORTLAND CEMENT MANUFACTURERS, LTD.

ALTHOUGH its manufacture is essentially under the guidance of the chemist, the true chemistry of Portland cement is far from being entirely fathomed. This lack of knowledge is not due to absence of research work, because the chemistry or mineralogy of cement has been the subject of investigation throughout the past century.

All are agreed that Portland cement contains certain proportions of silica, alumina and lime with other minor constituents, but there is no general agreement as to the precise manner in which these individual substances are combined. This limitation of fundamental knowledge, however, does not detract from the value or certainty of stability of cement, because there are decades of experience available to prove that within certain limits of chemical composition, and with the application of certain physical tests, the standard of quality of Portland cement can be vouched for.

It is known that Portland cement contains di-calcium silicate and tri-calcium silicate, and also tri-calcium aluminate with certain combinations of lime and iron oxide, which have not yet been thoroughly investigated. Attempts to separate any of these compounds from Portland cement by means of selective solution or otherwise have not been successful. The chemist has therefore resorted to the other means of synthesis and prepared the pure compounds mentioned, and has to a limited extent examined their properties. The preparation of these pure compounds has, however, been a matter of considerable difficulty because it has been necessary to employ the electric furnace and in some cases the temperature of the electric arc has been too high and has caused decomposition of the compound. Some therefore still doubt whether the compound tri-calcium silicate actually exists, and there are some experts who consider that any proportion of lime above that needed for the formation of di-calcium silicate is merely dissolved in that compound, forming a so-called solid solution. The absence of really conclusive information is so marked that there are several schools of thought possible in this connection, and debates still occur as to whether tri-calcium silicate actually exists in Portland cement or whether Portland cement is in reality a solid solution of one compound with another.

An important factor in the discussion upon the constitution of Portland cement is the limitation of the proportion of lime to silica and alumina which differentiates between a good and bad cement. Experience indicates that as the molecular ratio of lime to silica and alumina approaches 3.0 the cement improves in strength, but when it equals this figure the preparation of the raw materials calls for even more care, thus suggesting that there is an excess of lime beyond the tri-molecular proportion which is not so readily absorbed, and might cause unsoundness unless the raw materials are very finely ground. This is a strong argument

in favour of the theory that the essential constituents of a good Portland cement are tri-calcium silicate and tri-calcium aluminate, and it is one acted upon by the manufacturer whose aim is to increase the proportion of lime in his cement to this ratio in order to gain maximum strength. It is possible to raise the lime ratio without entailing unsoundness provided extreme care is taken in the grinding and mixing of the raw materials and in the burning of the cement.

So far as the solid solution theory is concerned it will be evident that hard burning is necessary before the degree of incipient vitrification is attained which permits of diffusion of the compounds into a mineral solution, so that in this connection the solid solution theory receives support from the known value of hard burning when dealing with high limed cements.

Research work upon the fundamental constitution of Portland cement is being continued in many quarters, and in the course of years some agreement will no doubt be reached as to the essential constituents, but it does not necessarily follow that such information when it is available will permit of better cement being produced with the raw materials at the manufacturers' disposal.

The investigation has, however, been led in another direction by some investigators who have borne in mind that as soon as cement comes into contact with water the high-limed compounds are broken down into lower-limed compounds, and it is the crystallisation and colloidal drying of these latter which actually causes the setting and hardening of cement. It has therefore been asked why, if these low-limed compounds are those which actually harden, should it be necessary to make the high-limed compounds, and why the low-limed compounds cannot be made in the first instance and in some way activated so that their crystallisation and colloidal actions take place on mixing with water. The use of lower-limed compounds in the manufacture of Portland cement would be a means of appreciable reduction in the cost, and it has often been attempted on an experimental scale. The results have, however, never been satisfactory in regard to strength.

Determination of Uncombined Lime.

Various methods have from time to time been suggested for the determination of uncombined lime in Portland cement, and it is of interest to know that in a paper on the subject issued from the United States Bureau of Standards in January, 1929, a revised procedure was recommended as a result of experience gained in the United States. The paper is a very lengthy one and is supplemented with numerous tables. Shortly, however, the determination is based on the solution of uncombined lime (CaO) in a hot solution of glycerol and alcohol, and the subsequent titration of the dissolved lime with an alcoholic solution of ammonium acetate in accordance with the reaction expressed in the formula

$$CaO + 2CH_3COONH_4 = (CH_3COO)_2\,Ca + H_2O + 2NH_3$$

The method of carrying out the determination is as follows: The cement is ground to an impalpable powder, and one gramme is weighed into a 200 c.cs. Erlemeyer flask containing 60 c.cs. of glycerol alcohol solution. The flask is well shaken, a reflux condenser attached, and the mixture boiled for twenty

minutes; the condenser is then removed and the solution titrated immediately with 0.2 N alcoholic solution of ammonium acetate. The condenser is then replaced, the solution again boiled for twenty minutes, and the titrations repeated at twenty-minute intervals until no further colour appears after boiling for one hour. The uncombined lime content of the cement is then calculated from the known lime (CaO) value of the standard alcoholic ammonium acetate solution used.

Throughout the determination it is essential that anhydrous alcohol be used, and the glycerol should be pure and free from moisture. The glycerol alcohol solution is one consisting of one part glycerol to five parts alcohol; this has been found to be the most satisfactory, as when the proportion exceeds 1 to 3 some of the compounds of cement are dissociated, and during boiling some concentration of glycerol may occur. To each litre of this solution are added 2 c.cs. of an indicator prepared by dissolving one gramme of phenolphthalein in 100 c.cs. of absolute alcohol. It is essential that the glycerol alcohol solution be just neutral to the indicator. The standard 0.2 N alcoholic ammonium acetate is made by dissolving sixteen grammes of crystalline ammonium acetate in one litre of absolute alcohol and standardised by titrating against pure freshly calcined calcium oxide.

During boiling care must be taken not to lose alcohol by too vigorous boiling and not to let carbon dioxide into the flask by not boiling vigorously enough, as in either case errors would be introduced. During titration great care must be taken to prevent ingress of carbon dioxide; therefore the solution should be as near boiling point as possible and the surrounding atmosphere free from the gas.

Chemical Analyses.

The chemical analysis of cements, if taken in conjunction with the mechanical and physical tests, affords much desirable information in the selection of a good quality material; indeed, it is almost necessary that both the chemical and mechanical examination of a cement should be made before the positive pronouncement of quality can be given, and when both have been ascertained the suitability and efficiency of a cement for any given purpose can be at once accurately determined.

In the testing of cements it is clear that in recent years there has been a substantial improvement in the application of well-known processes, such as the mechanical and chemical modes of evaluation, but there has been little alteration in principles.

As to the value of chemical tests, it is not definitely known just what part is played by each of the compounds that constitute present-day Portland cements, and chemical analyses do not explain the manner of the occurrence of these compounds. The definite aid which chemical analysis may render at the present time in determining the quality of a cement is therefore limited to ascertaining that the essential compounds are present in the proportions which experience

has proved to be necessary or most desirable for the cement in question, and to detect adulteration.

Neither complete nor partial chemical analysis of the constituents of hydraulic materials can therefore be ranked as infallible tests, or tests of primary importance. But chemical analysis may render real service in controlling the classification of a product concerning which there is reason to doubt the declaration of the manufacturer. Thus a slag cement can be distinguished from a Portland cement by its composition, as also may certain natural cements.

The chemical constituents of a well-made sample of Portland cement may be somewhat as under:

	Per cent.
Loss on ignition	1.22
Silica (SiO_2)	21.44
Insoluble residue	0.20
Alumina (Al_2O_3)	6.75
Oxide of iron (Fe_2O_3)	3.17
Lime (CaO)	63.44
Magnesia (MgO)	0.99
Sulphuric anhydride	1.81
Potash and soda	0.98
	100.00

There is a change in composition of the materials during the burning stage which is almost inevitably in the direction of reducing the lime factor. This is caused by the impurities picked up during the calcining process, such as a slight mixture of kiln linings and the ash from the fuel used with the kiln. These additions are chiefly composed of silica and alumina, and they affect and alter the theoretical composition of the cement as calculated from the raw materials used, by slightly increasing the percentages of silica, iron, and alumina, and reducing the lime content.

Effect of Fuel Ash.

The effect of fuel ash on the composition of cement as shown by analysis is illustrated by the following. The ash of the coal fuel other than oil used for the calcination of the raw material, being non-volatile, remains with the clinkered mass either wholly or in part according to the type of kiln used. Part of it is fused on to the outside of the pieces of cement clinker and combines with it, and part may remain as a more or less insoluble powder or clinker chiefly consisting of silicates of alumina and iron. Such ash as is not carried away by the draught in the form of dust is inseparable from the cement clinker, and is ground up with it. This obviously must affect the composition of the clinker. In the types of fixed kilns adopted formerly practically all the ash was thus left. Intermittent kilns required about 8 cwt. of coke to calcine a ton of cement, and continuous shaft kilns about 4½ cwt.

If we assume coke to contain 10 per cent. of ash, this means that 0.8 cwt. of ash was left in the first case and 0.45 cwt. in the other. This ash may have the following composition :

	Per cent.
Silica	51.0
Alumina and iron oxide	34.2
Lime	12.0
Undetermined	2.8
	100.0

and will affect the analysis of the average of the mass drawn from the kiln by reducing its percentage of lime. If in the raw material mixture the percentage of carbonate of lime be 76, and there be no volatile matter present other than the carbon dioxide belonging thereto, the amount of lime in the calcined mass, if calcined by an ashless fuel, would be 63.94 per cent. If calcined in an intermittent kiln under the conditions outlined already, the amount would become 61.86 per cent., and if calcined in a continuous shaft kiln 62.77 per cent.

Silica, Lime and Alumina.

The percentages of silica, alumina, oxide of iron, and lime contained in cements may vary considerably and still constitute a good and sound article, but magnesia and sulphuric anhydride should never greatly exceed the percentages given in the British Standard Specification.

Silica, lime, and alumina must be considered the essential elements of the cement as they combine in the kiln at a clinkering temperature to form compounds having special properties in relation to the effect that water has upon them. These compounds—silicate of calcium and aluminate of calcium—as they come from the kiln, and until they are wetted to form concrete, are perfectly anhydrous ; that is, they have no water chemically combined in their molecules. They are, however, capable of forming a chemical combination with water, and in that condition are spoken of as hydrated silicate of calcium and hydrated aluminate of calcium. The peculiarity of these compounds is that when in the anhydrous condition they are more soluble in water than when in the hydrated condition, the effect being that when wetted with water to form concrete the anhydrous substance is split up and a portion dissolves in the water. It then slowly combines with the water and the undissolved portion to form hydrated compounds, which, being insoluble, bind the whole mass into a solid block. As the solubility is but slight, this reaction takes place slowly, and it may be weeks or even months before it is complete. The property is known as hydraulicity, and a hydraulic substance is one that will set under water.

The great aim of the cement maker is therefore to produce the maximum amount of these substances in cement. If, on the one hand, there is too much lime in the mixture a portion of it remains uncombined and is a source of danger to the cement, as free lime expands and crumbles to dust on being hydrated (or "slaked" as it is commonly called), thus injuriously affecting the strength

and stability of the concrete ; so, on the other hand, if there is too much silica and alumina in the mixture, other compounds are formed which have no hydraulicity.

Lime Content.

Lime expands in slaking, so that an excess of lime above what will unite with the silica and alumina will cause the cement to expand, or " blow " as it is often called. If the percentage of lime is under the limit, the cement contains silica and alumina in excess, tending to produce weak cement. Unsoundness in cement is also caused by uncombined or loosely combined lime resulting from coarse grinding or irregular mixing of the raw materials or under-burning of the clinker, and this is detected by soundness tests.

Chemical analysis, therefore, if taken alone as the guide to quality, will seldom explain fully where unsoundness is concerned. Recourse must be had in this respect to the physical tests for soundness.

A Portland cement containing high percentages of lime requires high temperatures in the kiln whilst burning the raw materials. The rotary kiln has much improved the facilities for burning cement, and has resulted in a general increase in the percentage of lime content in cements.

An increase in the quantity of silica in cements requires a proportionate increase in the lime content with the result that the raw materials must be burnt at a higher temperature, and the resultant cement will be of a high tensile strength. Silica is generally contained in cements to an extent of 18 to 25 per cent. It is one of the most useful constituents, but must not be in insoluble form. Silica in Portland cement plays the part of an acid and combines under great heat with lime.

The function of the oxide of iron in cements, generally from 2 to 5 per cent., is to act as a flux in the kiln and cause the silica and alumina to combine with the lime at a lower temperature than they would without its intervention. At the same time the oxide of iron enters into combination forming similar compounds to the alumina compounds, and generally speaking the compounds of iron and alumina are interchangeable ; the difference is that whereas iron oxide lowers the temperature of burning and imparts the dark grey colour to cement, alumina makes for much higher burning temperature. Cements made with no iron are white.

White Portland cement is of similar character to ordinary grey Portland cements except that iron is almost entirely absent. For many of the purposes for which white cements are used, it is not the strength at early ages so much as the appearance of the finished product or structure which is of prime importance, and as this cement is hydraulic it can be used for exterior work, which was impossible with white cements and plasters produced from gypsum, such as Keene's cement.

The amount of alumina in Portland cement is generally from 3 to 10 per cent., and its function is assumed to affect the initial hardening of the material.

A high alumina content, for instance, makes a quick-hardening cement, and a low percentage of alumina in cements generally suggests slow-hardening qualities. Alumina in Portland cement acts as an acid.

Magnesia is present in cements up to about 3 or 4 per cent., a limit which is considered to be harmless ; indeed some experts consider that magnesia replaces lime in a cement, and this theory is growing in favour, although other experts have maintained that an excess of magnesia may lead to delayed unsoundness.

To ascertain the insoluble residue (such as sand, clay, and inert materia of any description) in a cement is useful in checking the existence of any foreign matter or adulteration in the product, as a pure Portland cement should contain

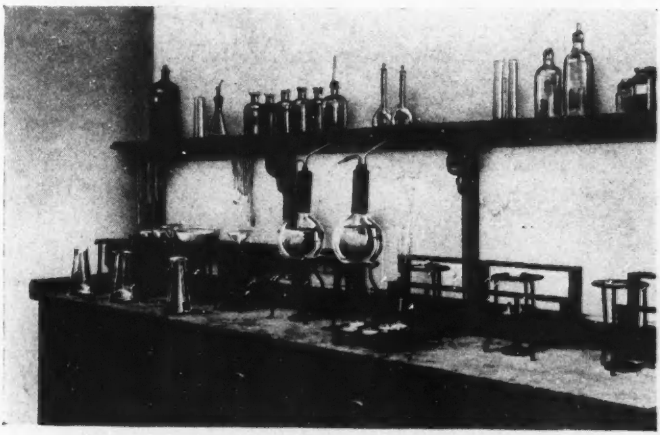


Fig. 1.

considerably less than 1.0 per cent. of such substances, which normally consist chiefly of detritus from the kiln and mill linings and uncombined fuel ash.

Analysis of Portland Cement.

There is no British Standard method for the analysis of Portland cement, and chemists may vary in their estimation of its constituents.

A typical working bench is shown in Fig. 1, and the methods usually adopted for analysing Portland cement are conducted as follows :

Weigh one gramme of the sample (Fig. 2) into a porcelain dish, swill round with a few c.cs. of hot water, add 10 c.cs. of strong hydrochloric acid, see that all lumps of cement are broken up and acted upon by the acid, cover the dish with a clock glass and evaporate to dryness on a hot plate (Fig. 3), carefully at first to avoid spitting ; then bake for one hour at full heat of plate. Remove, and when dish is cool enough to handle, add 25 c.cs. of strong hydrochloric acid ; leave for 15 minutes, warming very gently if necessary, until all red colour of the

iron salts has disappeared. Dilute with water, filter off separated silica and insoluble residue, wash well, dry, burn off, and weigh.

Ferric Oxide and Alumina.

The solution from the silica determination, having been collected in a suitable beaker, is washed back into the porcelain dish, heated to boiling, a few c.cs. of bromine water added, then 50 per cent. solution of ammonia added very slowly

Fig. 2.

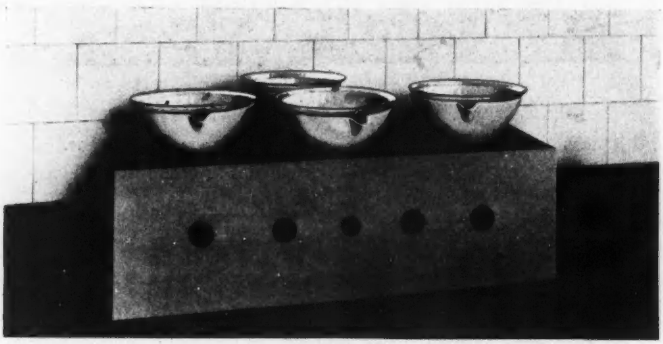


Fig. 3.

until the solution is neutral, then 2 c.cs. in excess are added, and the mixture boiled for two minutes. The precipitate is filtered off, washed twice and then washed back into the dish, dissolved in a few c.cs. of hydrochloric acid, precipitated as before, and filtered, collecting the filtrate in the same beaker as before. This precipitate should be well washed and then dried, burnt off and weighed.

FERRIC OXIDE.—A separate portion of one gramme is weighed into a porcelain dish, dissolved in hydrochloric acid and water, heated to boiling, and reduced with stannous chloride. When cool, 15 c.cs. of a saturated solution of mercuric chloride are added, allowed to stand for 10 to 15 minutes, and titrated with a standard solution of potassium dichromate.

LIME.—To the combined filtrates from the double precipitation of the iron and alumina, add 30 c.cs. of 50 per cent. solution of ammonia and bring to boiling, then add 40 c.cs. of a saturated solution of ammonium oxalate, boil for 5 minutes,

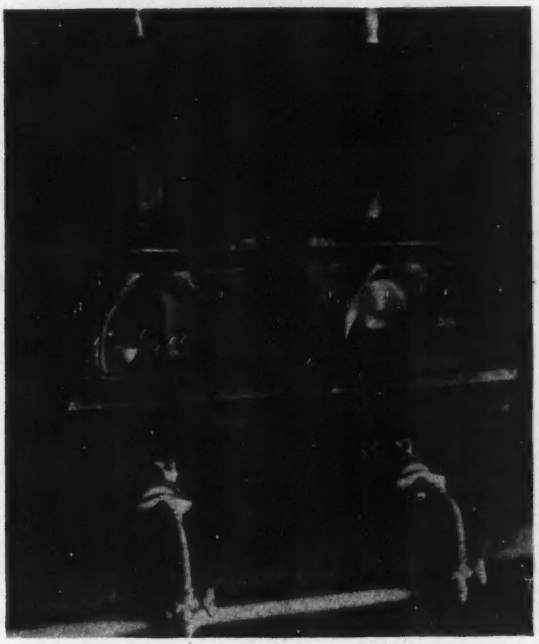


Fig. 4.

and allow the precipitate to settle. Decant as much of the clear solution as possible through a filter paper, keeping the precipitate in the beaker, wash two or three times by decantation. Now dissolve the precipitate in dilute hydrochloric acid and boil. Reprecipitate with ammonia and ammonium oxalate, and allow the precipitate to settle. Filter through the same filter paper, allowing the filtrate to mix with the first one. Wash the precipitate with warm water, thoroughly dry, burn off in platinum crucible, ignite gently at first until all carbon is burnt off, then strongly ignite in the muffle furnace (Fig. 4) until the weight is constant. The calcium oxalate is thus reduced to oxide and weighed as such. The method of filtering a lime precipitate is shown in Fig. 5.

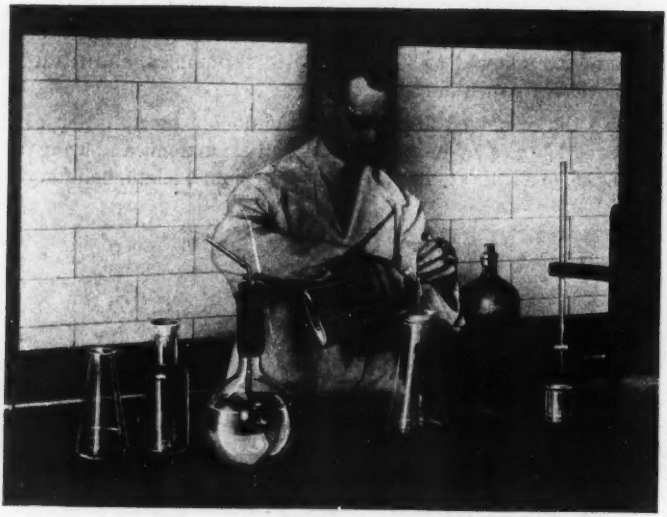


Fig. 5.

Fig. 6.

Magnesia.

The filtrations from the lime are evaporated to small bulk by boiling down in a porcelain dish. When the liquid has been reduced to as small bulk as possible on the burner, it is taken to complete dryness on the hot plate with the addition of 50 c.cs. of strong nitric acid to destroy the ammonium salts. When cool, take up with dilute hydrochloric acid, neutralise with ammonia, and filter off any precipitate. An excess of ammonium phosphate solution is added, viz., about 5 c.cs. of a saturated solution, together with about 20 c.cs. of strong ammonia, and the solution stirred briskly for about five minutes and allowed to stand over night. The precipitate is then filtered off and washed with cold dilute ammonia solution (10 per cent.). The precipitate is then dried (Fig. 6), ignited (first gently, afterwards in the muffle) and weighed as magnesium pyro-phosphate.

Sulphuric Anhydride.

Weigh one gramme of the sample into a porcelain dish, dissolve in hydrochloric acid and water as before, boil for 5 to 10 minutes to ensure all going into solution that will filter, and wash well. The filtrate is caught in a suitable beaker, raised to boiling, 10 c.cs. of a solution of barium chloride added, and the whole boiled for a few minutes. The precipitate of barium sulphate is allowed to settle over night in a warm place, filtered, washed well with hot water, dried, ignited and weighed.

Insoluble Residue.

The insoluble portion from the above is washed back into the porcelain dish, 20 c.cs. of a saturated solution of sodium carbonate added, boiled for 10 minutes, filtered, washed thoroughly with hot water, dried, ignited and weighed.

Loss on Ignition.

One gramme of the sample is weighed into a platinum capsule, placed in the front of the muffle furnace, left for 20 to 30 minutes, cooled and weighed.

Sulphur as Sulphide.

Weigh 2 grammes of the sample into a porcelain dish of suitable size, add a crystal or two of potassium chlorate, dissolve in 25 c.cs. aqua regia, evaporate to dryness, heat until dehydration is complete, take up with hydrochloric acid and water, and filter off silica in usual way. In the filtrate, determine the sulphuric anhydride by precipitation with barium chloride. This will give the total sulphuric anhydride, and the sulphur present as sulphide will be given by deduction of the sulphuric anhydride determined as such in the ordinary way, and a calculation to convert sulphuric anhydride into sulphur.

Potash and Soda.

Weigh 5 grammes of the sample into a platinum dish, dissolve in hydrochloric acid and water, evaporate to dryness, bake, take up with acid, and filter off silica in usual way. In the filtrate, precipitate sulphuric anhydride with barium chloride, after standing in warm place for few hours without filtering, add ammonia and ammonium carbonate in excess to precipitate alumina, ferric oxide, lime and barium; filter off all together and wash well; redissolve the

massed precipitates in minimum quantity of hydrochloric acid, reprecipitate with ammonia and ammonium carbonate, filter and wash well. Take combined filtrates to dryness in platinum dish; burn off ammonium salts. Take up with a few drops of hydrochloric acid and water, add ammonia and ammonium carbonate to ensure all lime, etc. (if any), is precipitated, filter off, evaporate to dryness, burn off ammonium salts as before, and repeat until free from all lime, etc. Next, add ammonium carbonate, an equal volume of alcohol, allow to stand for not less than 2 hours, filter off precipitate (some lime, chiefly magnesia), evaporate filtrate to dryness, burn off ammonium salts, take up with water, filter, add 2 or 3 drops of hydrochloric acid to filtrate, evaporate to dryness in platinum dish, burn off any ammonium salts, and weigh as the mixed chlorides. Dissolve the mixed chlorides in water, add an excess of platinic chloride solution.

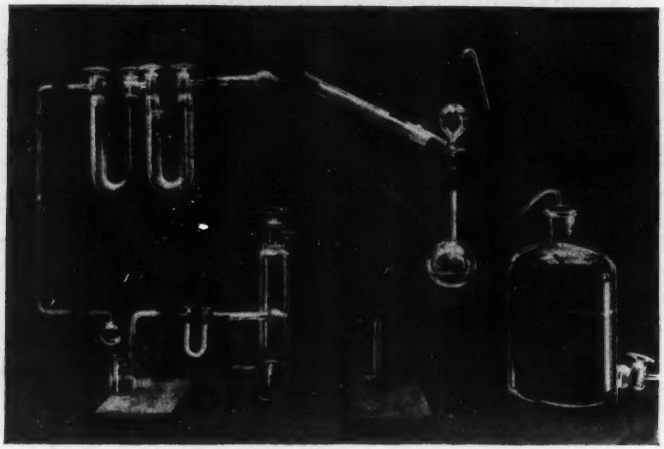


Fig. 7.

Evaporate nearly to dryness, add 20 c.cs. of 80 per cent. alcohol, and allow to stand until the sodium salts dissolve. Filter through a small filter paper and wash with 80 per cent. alcohol until washings run through perfectly colourless. Dry the filter paper and dissolve precipitate on it by washing with hot water, allowing washings to run into weighed platinum dish; evaporate off water, dry at 135 deg. C., and weigh as potassium platinic chloride. A calculation then gives the soda and potash.

Carbon Dioxide.

Weigh 5 to 10 grammes, according to the amount present, into a 250 c.cs. round-bottomed flask and connect to the apparatus (Fig. 7); aspirate for one hour to ensure the apparatus is free from contamination by carbon dioxide from the atmosphere. Next connect the weighed potash absorption bulb and guard tube and put 20 c.cs. strong HCl in the bulb of dropping funnel in neck of

flask, having previously closed all taps ; turn aspirator on full and open the taps one by one from the aspirator end, seeing that apparatus is gas-tight after opening each tap. Then very slowly open the tap in the bulb containing the acid, just sufficient to cause an evolution of gas through the potash absorption bulb at the rate of one bubble per second. When evolution of gas is complete, open full the tap controlling the acid, and adjust the aspirator to maintain correct rate of bubbling through potash absorption bulb ; turn on water to condenser, and apply heat to the flask ; heat to boiling, and boil for 10 to 15 minutes, after which continue aspirating for one hour to ensure complete absorption of all the carbon dioxide which has been liberated from the sample. Remove the potash absorption bulb and guard tube. Leave in balance case for half an hour to get to atmospheric temperature, then weigh. Increase in weight represents the carbon dioxide.

The results calculated to percentages are then tabulated as follows :

	Per cent.
Loss on ignition (water, carbon dioxide and other loss)	
Silica (SiO_2)	
Insoluble	
Ferric oxide (Fe_2O_3)	
Alumina (Al_2O_3)	
Lime (CaO)	
Magnesia (MgO)	
Sulphuric anhydride (SO_3)	
Alkalis (potash and soda)	
	100.00

Book Review.

The Chemistry of Portland Cement from the Point of View of Complex Chemical Compounds. By Lennart Forsen. (Berlin : Zementverlag).

This paper should be read with C. R. W. Mylius' " Calcium Aluminates and their Double Salts," reviewed in the May number of this journal. The author bases his theory of their constitution on A. Werner's work on inorganic compounds. He compares the various salts containing alumina with the complex cobalt and chromium compounds. Structural formulæ for the aluminates are developed and in some cases compared with well-known inorganic compounds. For instance, tetracalcium aluminate is likened to tetracalcium phosphate (isoclase). After the simple aluminates the double salts are dealt with and a set of photomicrographs is given.

Besides the aluminates there are a few pages on the basic calcium salts such as $3CaO.CaX_2.15H_2O$ where X may be Cl, Br, I, or ClO_4.

The hydrated silicates are described briefly, and the process of hydration of tricalcium silicate is followed by means of structural formulæ. The hydration is considered as taking place in three steps: $3CaO.SiO_2 - 2CaO.SiO_2.H_2O - 3CaO.2SiO_2.3H_2O - CaO.SiO_2.2H_2O$.

Quick setting is due to a reaction between the silicates and aluminates. Substances forming double salts with aluminates remove these from solution and hence slow the setting process. The chief reactions in setting are :

(1) $3CaO.Al_2O_3 + 6H_2O = Ca_3[Al(OH)_6]_2$ (in solution)

(2) $Ca_3[Al(OH)_6]_2 + CaX_2 = 2Ca_3[X][Al(OH)_6]$ (cryst.)

(3) $Ca_3[Al(OH)_6]_2 + Ca(OH)_2 = 2Ca_3[OH][Al(OH)_6]$ (cryst.)

(4) $Ca_3(SiO_3) + 2H_2O = Ca(OH)_2 + Ca_2H_2(SiO_4)$ (cryst.) (gel)

An important effect of hydrated calcium aluminate is that it forms nuclei for the crystallisation of calcium hydroxide. The strength of Portland cement depends upon the amorphous silicate gel and the crystallised calcium hydroxide, the crystallisation of which has been promoted by nuclei of calcium aluminate or basic calcium salts.

Steel Progress and the Cement Manufacturer.

[CONTRIBUTED.]

IN the urgency of concentrating on cement manufacture, and of keeping abreast of technical developments in his own sphere, it is difficult, if not impossible, for the cement manufacturer to follow every detail of importance to him in the evolution of steels. Nevertheless it is essential that he should take into account the advances made, as these have an influence upon the development of his plant. This article summarises the progress of the last year or two in steel and steel products as they affect cement works.

Chrome Alloy Steel.

Perhaps the most interesting advance of recent date is the introduction of a special chrome alloy steel primarily intended for those parts of crushing and grinding machinery for raw materials that have to withstand severe wear, but which should be free from the spreading action under repeated hammering that is one of the disadvantages of high manganese steels. This new steel is particularly to be recommended for lining plates of tube mills, rod mills, ball mills, etc., for the spiders of rod mills, the diaphragms and end-plates of tube mills, for the rolls of crushing rolls, and many similar purposes. More than one grade of this steel is available according to the duty required, the grades varying in chemical composition.

As a rule castings made from these special steels are given a double heat-treatment, resulting in a better physical condition. As compared with similar castings of high manganese steel they cost somewhat less, and, owing to their high elastic ratio, they do not flow sideways under percussion as happens with high manganese steel. Further, it is not so difficult to machine the castings, which is an advantage even if only slow cutting speeds can be employed. The main requisite, namely high resistance to wear, is extremely good, and a Brinell of 255 can usually be relied upon for lining plates made from this special chrome steel.

Light Castings.

A second development of significance to the cement manufacturer is the successful introduction and establishment of a new method of manufacturing small and light steel castings, particularly those employed in crushing and grinding machinery. The feature of this process is that it reduces the machining allowance required by the steel founder, and as a result it is possible to lessen the weight of the castings. In addition, as there is not so much metal to take off, machining becomes less expensive, while jigs can be made use of in course of production because the castings are so true to shape. The benefit to the purchaser is a saving of money, since losses due to defects not being discovered before the machining operation has begun are eliminated. Light and intricate castings of the kind outlined can now be bought in large numbers with speedy delivery and perfect soundness, even though they weigh as little as a few ounces.

Working High Manganese Steels.

An important discovery is the production of super-high-speed steels capable of commercially working and drilling high manganese steel. Hitherto the machining and drilling of manganese steel was, from a commercial point of view, impossible. When holes were required in manganese steel castings for crushing and grinding or similar machinery, they had to be cored in and finished to size by grinding, a slow and costly operation. Now it is possible to bore, drill, turn, plane, etc., high manganese steel as a commercial proposition, and the cost of production of these castings has been reduced as a consequence while delivery has also been speeded up.

The coming of the electric high-frequency crucible furnace, and the cobalt steels produced in it, have alone made this feasible. As an example of the effect upon the cement industry, it is now possible to prolong the life of worn crushing rolls made from high manganese steel. The teeth of these rolls invariably wear down first in the centre of the roll, and the roll then becomes of little value. The outer and less worn teeth can now be reduced by machining with super-high-speed steel until they are of the same height as the more worn teeth. Consequently the roll can be put back into service, and the operation repeated until all the teeth are uniformly unserviceable.

Similarly the drilling of holes in manganese steel is now feasible, but for this work the drills have to be of special design and a proper, though not difficult, drilling technique must be followed.

Fine Tool Steels.

The high-frequency crucible furnace referred to earlier has revolutionised the Sheffield steel-making industry so far as fine tool steels are concerned. Previously all fine cutting and similar steels were made in crucibles heated in gas-fired or coke-fired furnaces and teemed out into ingot moulds by hand, a severe and physically taxing operation. Under the new process the steel is melted by eddy currents set up in the steel by high-frequency current. There is no contact with coke, gas impurities, or even with carbon arcs (as in the electric arc process), and consequently a pure steel is produced. Further, a severe electrical stirring is set up in the molten metal by the high-frequency current, and this completely diffuses the heavy alloying elements (cobalt, tungsten, molybdenum, and the like) throughout the entire mass. The normal tendency of these elements to sink to the bottom of the pot is thus counteracted, and as a result a homogeneous steel is produced. The other advantages of this new electrical process are technical, and need not be described here.

Heat Resistance.

The development of rotary kilns is to some extent linked up with the production of a variety of steels of highly complex composition, all offering very high resistance to heat and to the scaling caused by heat. One main group of these recently developed steels has in its composition percentages of such alloys as nickel, chromium, copper, tungsten, and silicon. With these steels heat resistance up

to 1,200 deg. C. can be obtained with a diminution of mechanical properties (tensile strength, etc.) very much less than would be the case with ordinary steels. In addition, such steels offer a high resistance to corrosive action.

A chrome-tungsten heat-resisting steel is one of the most recent to be placed on the market, and has been evolved primarily to give a " creep " as low as possible at high temperatures while retaining maximum strength. Another heat-resisting steel has a high percentage of chromium, together with silicon or aluminium or nickel. This steel offers extreme resistance to oxidation.

Drills.

In rock and similar drill steels for quarry work, boring through rock, minerals, and other hard substances, the primary development has been in connection with hollow steel, and various improved methods of forming the hole have been introduced. In one of these improved methods the steel billets are rolled with an austenitic steel core which is afterwards extracted. The result is a hole whose surface is clean, smooth, and free from irregularities. The importance of the surface of the hole lies in the fact that it is usually the starting point of fine cracks, which spread until they lead to the failure of the drill in use, so that the smoother and freer from irregularities this surface is the less likely is the steel to crack. In other methods the billets are rolled on copper or sand cores.

Drilling has not shown any great advance on former methods so far as the steel drills are concerned. One must mention, however, the experiments being made with detachable bits. Many users consider these offer important advantages, but opinion is sharply divided on the point. For the detachable bit it is claimed that it is easier to handle; that smaller stocks of steel can be kept, thus reducing handling charges and saving time; that it is possible to employ differently composed steels for bit and shank, thereby increasing efficiency; that a bit can be supplied which will work more quickly and retain its cutting edge for a longer period than can be done with the solid drill, while at the same time a shank of a different steel, chosen specially for its toughness and length of life, can be employed; that the sharpening and production of the bits can be centralised and greater uniformity is attainable, while the bulk of steel needed is smaller. On the other hand, it has been stated that in practice these advantages would not materialise, and that no entirely satisfactory method of fitting bit to shank has yet been found.

Spades and Shovels.

While on the subject of quarry work it may be of interest to note the introduction of an alloy steel of special composition for spades, shovels, and kindred tools. While the normal steel-bladed shovel is satisfactory for general purposes, it often happens that a shovel or spade with greater wear-resistance is required. A special steel is being produced for tools of this kind, and contains roughly 0.40 carbon, 0.60 manganese, 1.25 nickel, 0.80 chromium. Shovels and spades manufactured from this steel are generally hammered out from the solid bar instead of being cut from plain sheets. It is claimed for them that they are light, strong, tough, and so pliant that they will bend in a vice but immediately

spring back to normal when freed. They are said not to break in use, and not to turn up at the corners. Though their first cost is rather higher than that of ordinary spades and shovels, they last from three to five times as long and even longer life can in most instances be obtained.

Manganese Steel.

So far as materials for the construction of cement-making machinery are concerned, only the special chromium steel has so far been referred to. In manganese steel there have been no vital changes, but some foundries are discovering that higher mechanical properties are obtainable by raising the manganese content and lowering the carbon.

Nickel Steel.

But there have been developments in steels for cast parts of other machinery, such as excavators. Here, nickel is being increasingly employed as an alloying material in racks and pinions, gearing, runner wheels, etc. It has the effect of raising the toughness of the castings, refining the grain, and at the same time enabling the steel to respond with greater facility and effectiveness to heat-treatment of suitable character. Exceptional resistance to abrasive action and wear is demanded of tractor, excavator, and other machinery parts, while high-tensile strength plus the ability to resist severe and sudden shock are also needed. Racks and pinions for excavators are now being manufactured in many foundries from nickel chromium and nickel-chrome-molybdenum steels, while similar steels are being used for the various gears and driving sprockets.

Welding.

In the large electrical machinery employed for motive purposes in cement works the motor-cases for the dynamos were formerly almost always manufactured from a special steel known as dynamo-magnet steel. In this steel there has been no change, but by far the larger number of these motor-cases and similar electrical machinery parts are now built up by fabrication. Sections of steel are welded together to form the complete body or other part required, and consequently a great cheapening in cost is rendered possible while the parts themselves are lighter, thus saving time and labour. The development in the welding of steel during the last few years has been enormous. One development that has already been suggested in more than one quarter, and that may materialise before long, is the complete welding together of kiln, etc., sections, which are at present joined by the laborious and costly process of riveting. This change is strongly opposed in more than one quarter, possibly on good grounds, though one is tempted to wonder if conservatism is not responsible to some extent. However that may be, it will certainly not be long before the experiment is tried, and possibly it is only the rather costly character of the experiment that has prevented its being tried before.

Enough has been written to show that the steel industry has by no means stood still, and that despite the blighting effect of the current world depression, research is still taking place, and will inevitably bring results as fruitful as, and perhaps even more fruitful than, those described in this article.

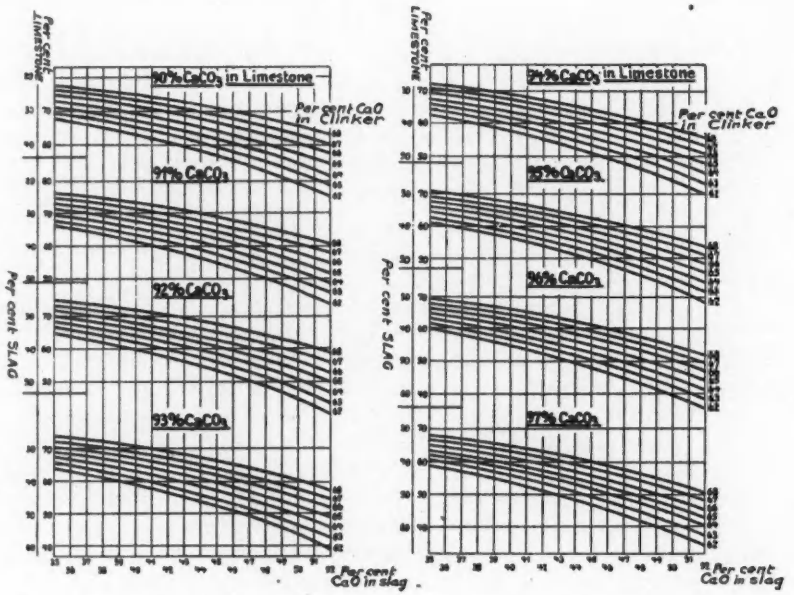
Nomogram for Mixing Raw Meal.

A METHOD is described and a nomogram given by F. Wenzel in a recent number of *Zement*, whereby the proportions of the cement raw materials (in this example limestone and slag) can be read from a curve, given the $CaCO_3$ content of the dry limestone (c), the CaO content of the dry slag (b) and the CaO content required in the clinker (i.e., the CaO content of the ignited raw meal, designated a). The nomogram is built up from the equations

$$x + y = 100.$$

$$x = \frac{100(a - b)}{c\left(\frac{z}{100} + \frac{ua}{10{,}000}\right) - b},$$

where a, b and c have the meanings stated, x = percentage of limestone, and y = percentage of slag in the raw mix, z = molecular weight of CaO and u = molecular weight of CO_2.



As an example, let $c = 95$, $b = 48$ and $a = 68$ per cent. Working with the curves for 95 per cent. $CaCO_3$, take the curve for 68 per cent. CaO in clinker (marked on the right of the diagram). Intersect this with the ordinate at 48 per cent. CaO in slag (marked on base of diagram), and for the point so obtained read the proportions on the left of the diagram ; the values obtained are limestone 59.5 and slag 40.5 per cent.

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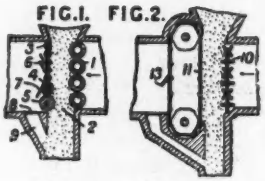
The nomogram is useful where the raw materials vary in composition. The examples shown range from 86 to 97 per cent. $CaCO_3$ in limestone, 62 to 68 per cent. CaO in clinker, and 35 to 52 per cent. CaO in slag. The curves may be extended for use with other raw materials.

Recent Patents Relating to Cement.

Portland Cements.

385,109.—Andreas, A., 3, Engelstrasse, Münster, Germany. Dec. 18, 1931. Addition to 360,547. [Class 22.]

In apparatus for preheating raw cement material comprising two spaced perforated walls or grids between which passes the material to be heated by waste gases from the kiln which flow through the grids, at least one of the grids is formed of separate rotary elements. Fig. 1 shows a construc-



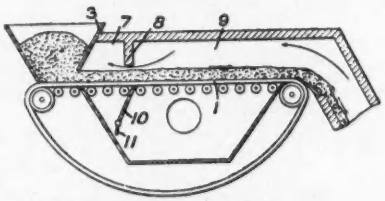
PORTLAND CEMENTS.

tion in which both grids are constituted by driven rollers, the front rollers (1) being of non-metallic refractory material and of uniform diameter, while those constituting the rear grid (3) are formed of iron and increase in diameter downwardly as shown at 4, 5. In the modification shown in Fig. 2, the front grid is formed of cruciform rotary elements (10) and the rear grid is constituted by an endless band (11) which may be formed either of perforated plates as shown or of inclined slats connecting a pair of endless chains. In this form the gas outlet may be arranged between the runs of the band (11) so that the outer flight thereof is disposed in the open air. Scrapers for cleaning the outer side of the rear grid are shown at 6, 7 (Fig. 1) and at 13 (Fig. 2), the removed material being returned to the column (2) through the valve (8) and the chute (9). A further construction is described in which both grids are similarly formed of hollow air-cooled rollers having radial perforations through which the cooling air escapes, these air jets serving to keep the grid openings clear.

Calcining Cement, Lime, etc.

386,330.—Trigga, W. W., 57, Lincoln's Inn Fields, London.—(Polysius Akt.-Ges., G.; Dessau, Germany), Lepol, Internationale Patentverwertungs Ges., 181, Friedrichstrasse, Berlin, and Naamlooze Vennootschap Solopol, Ingenieurs-Bureau Tot Exploitatie Van Het Systeem Polysius, 44, Java Straat, The Hague, April 9, 1931. Addition to 288,192 [Class 22].

Cement raw material, lime, etc., in the form of granules is, prior to calcination on a travelling grid or the like, as described in the parent Specification, dried under such conditions that the material is not exposed to a calcining temperature during the drying treatment, and breaking up or bursting of the granules is prevented. The drying may be effected by forcing through the material a mixture of exhaust gases from the rotary furnace mixed with cold air or with waste gases from the travelling grid. This may be effected by providing a separating wall between the charging hopper and the calcining chamber, projecting downwards and



CALCINING CEMENT, LIME, ETC.

leaving a space at the bottom for the entry of a limited quantity of drying gases. A slide or the like may be provided to adjust the opening, and the suction draught may be regulated by providing below the drying chamber a protection sheet and flap valve. In a modification the drying chamber is cut off from the calcining chamber. As shown, the granulated material from the hopper (3) is fed on to a travelling grid (1) on which it is first dried and then calcined. The drying chamber (7) is separated from the calcining chamber (9) by a wall (8) extending downwards, leaving a space at the bottom for the passage of gases. To control the suction draught a protecting sheet (10) and throttle flap (11) are provided beneath the drying chamber.

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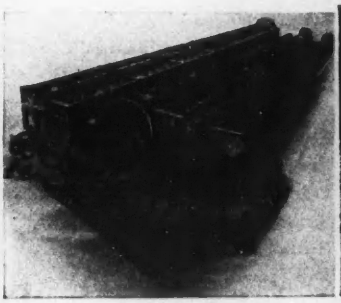

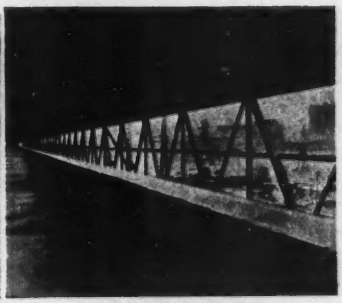

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